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(54) Title: CONNECTION WITH A COMPONENT FABRICATED OF A THERMOPLASTIC ELASTOMER

(57) Abstract

The invention relates to a process for the fashioning of a sealing connection between at least 2 components of an object, with at least one component being obtained by moulding a thermoplastic elastomeric material, characterized in that the thermoplastic elastomeric material exhibits at least 2 melting points and in that the component, on being fitted at the location of the desired connection, is exposed to a temperature which lies between the lowest and the highest melting point of the thermoplastic elastomeric material. Components that are suitable for application in the process of the invention also form part of the invention. The process is particularly suitable for producing body plugs that are applied in car body parts which subsequently are to pass a paint spraying line.

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CONNECTION WITH A COMPONENT FABRICATED OF A THERMOPLASTIC ELASTOMER

The invention relates to a component fabricated of a thermoplastic elastomer which is applied in a permanent connection between at least two components in an object and a process for the fashioning of this connection. The invention is particularly aimed at a process for bringing about a sealing connection between two components in an object.

Sealing components of thermoplastic elastomeric materials are known and find wide application, for example as a body plug, that is, a plug for sealing holes needed in the construction of, for example, a car body or refrigerator to prevent ingress of water, caps, sealing rings, shrink-on sleeving and the like. Application of sealing objects of thermoplastic elastomeric material presents important advantages in that the thermoplastic properties allow more complex mouldings to be mass-produced in a fairly simple manner, which is not possible in the case of rubber materials since, here, vulcanisation needs to take place in the mould. However, a disadvantage of the sealing or connecting objects fabricated of thermoplastic elastomeric material is that they allow only a limited degree of deformation when they are fitted. In general a degree of deformation of the order of some tens of percent is not completely reversible and results in permanent deformation.

If the initial deformation is 100%, permanent deformation often can be as high as 40%.

This is referred to as a tension set of 40%. In general, such tension set is less than 10% for rubbers. In some cases, performance is inadequate because of this higher tension set and also because thermoplastic elastomeric materials generally are harder due to the presence of hard segments in the matrix. Consequently, the object of the invention is to provide a process for the fashioning of a permanent connection between two components of an object, in which at least one component is obtained by moulding a thermoplastic elastomeric material, which results in an improved seal.

The object of the invention is achieved by the process whereby the thermoplastic elastomeric material of the one component obtained by moulding from the melt exhibits at least two different melting points and the component, on being fitted at the location of the desired connection, is exposed to a temperature which lies between the lowest and the highest melting point of the thermoplastic elastomeric material.

Thermoplastic elastomeric materials are described extensively in for example Thermoplastic Elastomers, 2nd impression, G. Holden e.a. editor, Hanser Verlag (1996), ISBN 1-56990-205-4, and the literature cited therein.

A thermoplastic elastomeric material having at least two melting points can be obtained in various ways. A simple method is the one whereby a higher melting thermoplastic elastomer is blended with a lower melting thermoplastic elastomer. Although it is preferred for the polymeric components of the thermoplastic elastomeric material having at least 2 different melting points to possess all elastomeric properties, the use of one or more polymers that do not possess these elastomeric properties as a component of

the elastomeric material for the process of the invention is not ruled out. In such case the non-elastomeric component preferably is the component having the lowest melting point.

The thermoplastic elastomeric material may contain common additives, for example fillers such as carbon black, talcum, reinforcing fillers such as mica, stabilisers, colorants and processing aids.

The choice of the components for the thermoplastic elastomeric material is dictated not only by the mechanical requirements but also, in part, by the medium to which the seal is exposed, the operating temperature and the temperature to which the seal is temporarily exposed in the process of the invention. One skilled in the art will make a choice from case to case on the basis of their knowledge of the physical and chemical properties of the various components.

If the sealing connection is used as a body plug, the temperature to which it is temporarily exposed will usually coincide with the temperature of the paint spraying line in which the car body part containing the seal is spray-painted. In that case, the duration of the temporary exposure will in general also coincide with the time it takes for the paint to be applied and to cure. This temperature usually is between 150 and 200°C and the duration of the operation between 10 and 2000 seconds.

In the above-mentioned case, the choice of the components of the thermoplastic elastomeric material needed in the process of the invention is adapted to the given conditions of the paint spraying line. In other cases, in which for example the chemical properties of this sealing material play an overriding role in the choice of the components, the temperature

to which the component is exposed in the process of the invention will be determined by the material selection. In the case of the body plug for car body parts, it is often a secondary requirement that the component must be well paintable. In that case a somewhat polar material is to be preferred. Particularly suitable in this respect are copolyether esters composed of hard polyester segments derived from alkylene diols and aromatic or cycloaliphatic dicarboxylic acids, for example polyethylene naphthalate, polypropylene terephthalate and polybutylene terephthalate and soft segments derived from alkylene oxide-based polyols such as ethylene oxide, propylene oxide and butylene oxide or combinations thereof. Use is preferably made of copolyether esters based on polybutylene terephthalate and polybutyleneoxide diol or ethylene oxide-terminated polypropyleneoxide diol.

The proportion of the component having the highest melting point (relative to the total proportion of polymer) will in general be more than 50 %wt. The proportion of the low-melting component is in general at least 1 %wt. , preferably 4 %wt. and most preferably at least 10 %wt.

It is preferred for the components to be so chosen that the melting points of the lowest melting and the highest melting components differ by at least 30°C; more preferably, this difference is at least 40°C and most preferably at least 50°C. In practice, a minimum difference of 10°C seems necessary for the techniques currently available.

The difference between the elevated temperature and the melting point of the lowest melting component is to be chosen depending on the time during which the component is exposed to the elevated

temperature. In general, this difference is between 10 and 50°C, with a greater temperature difference being preferred for a brief treatment and a smaller temperature difference being sufficient in the case of prolonged treatment.

During the exposure to the elevated temperature it may be advantageous to press the components that make up the connection onto one another with a mild force. In general, such force is so chosen that only minor deformation of the components that make up the connection can occur. One skilled in the art can establish this on a case-to-case basis.

Moulding of the component is not bound to any particular technique and may be effected by common techniques such as injection moulding and extrusion techniques.

Also covered by the invention is a thermoplastic elastomeric component suited to form a connection using the process of the invention.

A surprising additional advantage of the process of the invention and of the components suited for use in the process of the invention is that exposure of the components that make up the connection to the elevated temperature substantially nullifies the tension set, i.e. deformation, if any, occurring during the fitting of the components, so affording higher reproducibility of the seal quality. This is particularly the case with body plugs as used in for example car body components.

The invention is now elucidated by the following examples and comparative examples. It will be clear to those skilled in the art that the invention is not limited to the combinations of materials and conditions chosen but that these may vary from case to

case.

Where the description refers to melting point, this should be understood to mean the peak temperature in the 2nd heating curve as determined through differential scanning calorimetry (DSC) at a scanning rate of 20°C/minute.

Examples and comparative experiment

Materials used

- Arnitel PL 380®: a polyether ester block copolymer, Shore D hardness = 38 and melting point 220°C from DSM, the Netherlands
- Arnitel E 40 D®: a polyether ester block copolymer, Shore D hardness = 40 and melting point 150°C from DSM, the Netherlands
- Elvax Low Melt® ethylenevinylacetate copolymer with melting point 90°C.
- Sheet steel coated with a primer (epoxy resin with inorganic filler)
- galvanised steel sheet

Experimental

Granules of the various materials were blended in varying compositions that were subsequently injection moulded in an injection moulding machine provided with a single-screw extruder at a temperature of 235°C and for the rest identical injection moulding conditions in order to test specimens 2 mm thick. The various test specimens were contacted with the steel sheets for 30 minutes at 180°C. A mild pressure of

6 kN was applied in order to ensure proper contact. Next, adhesion between the specimens and the metal substrate was measured by means of the 90° peel test using a Zwick 1445 testing machine.

Sample dimensions l = 90 mm, w = 30 mm.

Table 1 presents the compositions tested and the measuring results obtained.

Composition	Adhesion on primed steel [J/m^2]	Adhesion on galvanised steel [J/m^2]	Notes
PL 380	0	0	Comp. Exp.
E40D	coherent fracture	333 (20)	Comp. Exp.
PL380/E40D 75/25 %wt.	100 (10) *)	266 (14)	
PL380/E40D 50/50 %wt.	33 (2)	200 (8)	some deformation
PL380/Elvax 96/4	15 (4)	22 (3)	
PL380/Elvax 75/25	133 (7)	200 (10)	
PL380/Elvax 50/50	333 (14)	166 (9)	some deformation

*) standard deviation

As appears from the above results, a very strong connection is brought about by the process of the invention between the (pre-treated) metal surface and the thermoplastic elastomeric component, which heavily contributes to a lasting seal and furthermore is insensitive to mechanical loads, for example shocks and impacts.

Thermoplastic elastomeric components suitable for use in the process of the invention also form part of the invention.

The Shore D hardness of the composition is not necessarily limited to approx. 40 but may vary from very soft to hard depending on the mechanical requirements of the sealing layer.

CLAIMS

1. Process for the fashioning of a sealing connection between at least 2 components of an object, with at least one component being obtained by moulding a thermoplastic elastomeric material, characterized in that the thermoplastic elastomeric material of the component obtained by moulding exhibits at least two different melting points and in that the component, on being fitted at the location of the desired connection, is exposed to a temperature which lies between the lowest and the highest melting point of the thermoplastic elastomeric material.
2. Thermoplastic elastomeric component suitable for application in the process of Claim 1.
3. Body plug suitable for the sealing of openings in car body parts, characterized in that the body plug is obtained by moulding a thermoplastic elastomeric material that exhibits at least 2 melting points.
4. Body plug according to Claim 3, characterized in that it is obtained from a blend of at least two copolyether esters having different melting points.

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B. FIELDS SEARCHED

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Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
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A	US 4 390 668 A (GARVER SR EDWARD B) 28 June 1983 (1983-06-28) claims	1,2,4
A	EP 0 834 442 A (UNITED CARR GMBH TRW) 8 April 1998 (1998-04-08) figures	1
A	US 5 829 482 A (TAKABATAKE YOSHIHIRO) 3 November 1998 (1998-11-03) figures	1

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Information on patent family members

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(54) **CONNECTION WITH A COMPONENT FABRICATED OF A THERMOPLASTIC ELASTOMER**

VERBINDUNG MITTELS EINES THERMOPLASTISCHEN-ELASTOMER ELEMENTES

RACCORD AU MOYEN D'UN COMPOSANT FABRIQUE A PARTIR D'UN ELASTOMERE THERMOPLASTIQUE

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US-A- 4 390 668
US-A- 5 829 482

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(54) **CONNECTION WITH A COMPONENT PRODUCED OF A THERMOPLASTIC ELASTOMER**

VERBINDUNG MITTELS EINES THERMOPLASTISCHEN ELEMENTES

SCELLAGE REALISE AVEC UN ELEMENT D'ETANCHEITE OBTENU A PARTIR D'UN ELASTOMERE THERMOPLASTIQUE

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(73) Proprietor: **DSM N.V.**
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- **DATABASE WPI Section Ch, Week 9231 Derwent Publications Ltd., London, GB; Class A23, AN 92-254019 XP002103263 & JP 04 170426 A (NIPPON ESTER CO LTD), 18 June 1992 (1992-06-18)**

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Inter nal Application No
PCT/NL 99/00554

C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

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Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	US 4 415 394 A (CHOLMAR SIDNEY) 15 November 1983 see the whole document	1-6,10
X	US 4 096 013 A (LUTZMANN H HARALD ET AL) 20 June 1978 see the whole document	1-6,10
A	US 4 365 716 A (WATT WILLIAM E R) 28 December 1982 see figure 1 see claims	1-10
A	US 5 532 053 A (MUELLER WALTER B) 2 July 1996 see column 7, line 42 - column 8, line 11 see claims	1-10

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[X] Further documents are listed in the continuation of box C. [X] Patent family members are listed in annex.

* Special categories of cited documents :

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Date of the actual completion of the international search	Date of mailing of the international search report
27 March 1998	09/04/1998
Name and mailing address of the ISA European Patent Office, P.B. 5818 Patentlaan 2 NL - 2280 HV Rijswijk Tel. (+31-70) 340-2040, Tx. 31 651 epo nl, Fax: (+31-70) 340-3016	Authorized officer Oudot, R

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